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HEAT PIPE DEVELOPMENT STATUS

by

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ABSTRACT

Test heat pipes have been operated in the 1400 K to 1700 K range for periods in excess of 20,000 hours with the objective of understanding and controlling corrosion and failure mechanisms. The results of a post test analysis of one of these heat pipes that was operated for 25,216 hours at 1700 K are reviewed and the implications for heat pipe lifetime discussed.

An in-process report of an investigation of transient heat pipe behavior is presented. This investigation is being conducted as a result of restart problems encountered during life test of a 2 m. radiation cooled heat pipe. The results of a series of shut-down tests from power and temperature are given and probable causes of the restart problem discussed.

INTRODUCTION

Heat pipe development work at Los Alamos National Laboratory has in the recent past been directed to the demonstration of the performance levels required for space power applications for both primary heat transport and heat rejection. In these investigations power levels of more than 19 kW/cm² axial flux density and 300 W/cm² radial flux density have been demonstrated at temperatures of 1400 to 1500 K, corresponding to primary heat transport conditions. Radiator heat pipe designs have been demonstrated at power levels of 3.1 kW per heat pipe with lengths to 5.5 m. Current investigations are directed to the identification of life controlling factors in high temperature liquid metal heat pipes and to the investigation of the behavior of high performance heat pipes under start-up, shut-down, and transient operating conditions. As a part of this investigation test heat pipes have been operated at temperatures in the 1400 K to 1700 K range for periods in excess of 20,000 hours. At test conclusion these life demonstration heat pipes have been subjected to detailed chemical and metallurgical post-test examination in order to determine the mechanisms of eventual failure. The objective of this work is the control of corrosion and failure mechanisms within the heat pipes in order to attain operating lifetimes of 7 to 10 years at high operating temperatures.

Recent investigation of transient heat pipe behavior at Los Alamos has been directed to the investigation of an evaporator dry-out condition

observed in the course of a high power extended performance demonstration of a lithium-molybdenum heat pipe. This test heat pipe was operated at high radiation load levels through a combination of high operating temperature and use of a high emissivity surface coating. The observed shut-down dry-out was felt to be a consequence of the test loading conditions, as no similar restart problem has been encountered in extensive testing of high power, high temperature heat pipes. The problem was felt to be of immediate interest to the SP-100 program, however, because these loading conditions are similar to those encountered in heat pipe application in radiation coupled primary heat transport systems. The extended performance test was therefore interrupted in order to investigate the phenomena. This investigation is continuing and the report herein constitutes an in-process status report rather than a complete resolution of the problems.

HEAT PIPE LIFE TESTS:

Test Hardware Description

Over the past years a series of sodium and lithium life test heat pipes have been operated at Los Alamos. These heat pipes are intended to be operated to failure and subjected to post test analysis to identify failure causes. The tests are continuing on 3 sodium and 3 lithium heat pipes operating in the 1400 K to 1500 K range. Of the heat pipes that have failed in test the longest period of operation was achieved with a molybdenum/lithium-configuration that was operated at 1700 K for 25,216 h. The heat pipe has now been analyzed in detail in an attempt to identify the failure mechanism. A cross section of the heat pipe is given in Figure 1. The outside envelope of the heat pipe consisted of a 165 mm long, low carbon arc cast molybdenum tube with an outside diameter of 25.4 mm. and a wall thickness of 1.78 mm. A

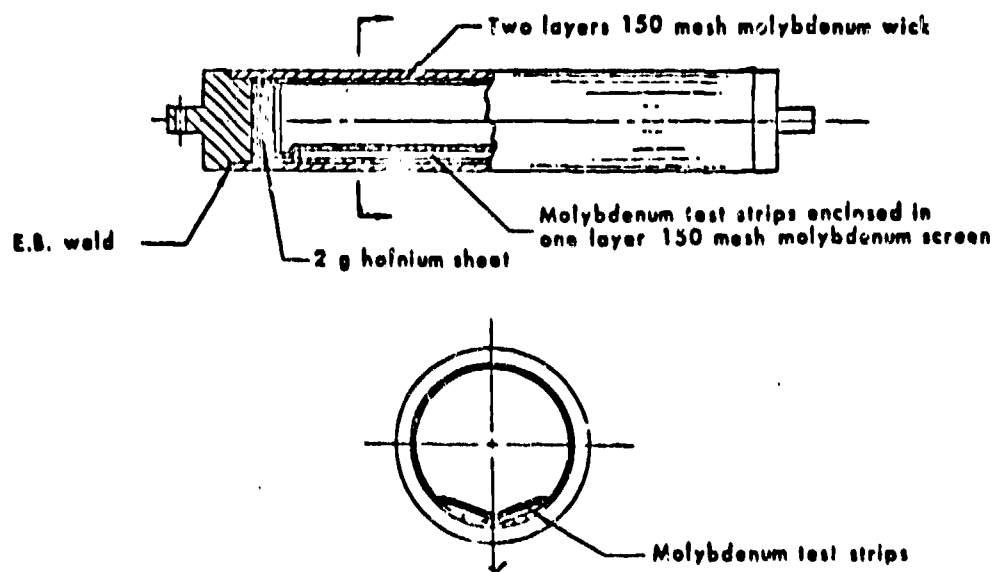


Fig. 1. Mo/Li heat pipe corrosion specimen.

two layer screen wick of 150 mesh molybdenum screen was used. Hafnium foil material was included in the heat pipe for gettering. Test strips of HT grade molybdenum were also included for compatibility determination.

This heat pipe was originally charged with lithium by placing a quantity of solid lithium sufficient to fill the wick plus some excess inside the container tube. The closure end-cap was then inserted into the open end of the container tube, and the heat pipe was operated in a vacuum to drive out the noncondensable gases and make a temporary vacuum-tight lithium seal. Final sealing was accomplished by electron beam welding the end cap.

Test Operations

For life testing the heat pipe was sealed in an evacuated quartz tube and heated by high frequency RF-induction over 5 cm of length near one end. The heat pipe was operated in a horizontal position with constant radial orientation. The temperature of the pipe was measured optically during the test. The total output power of the heat pipe at 1700 K was calculated to be 1056 W. Radiographs of the heat pipe were taken periodically during its lifetime and did not indicate any deterioration of the container tube. Periodic diametral measurements were taken on the container tube, with observed small increases close to that predicted from uniaxial creep data for molybdenum. During the total of 25,216 h of operation at 1700 K the heat pipe underwent at least 233 thermal cycles between room temperature and 1700 K. Failure of the heat pipe occurred through development of two small holes in the evaporator. Lithium loss from the holes caused the optical sensor to detect a malfunction and turn off the rf-generator. The quartz envelope was not broken in the shut down.

Post Test Analysis

During disassembly, the heat pipe was found to be mostly empty of the lithium, so post-test chemical analysis of the lithium working fluid could not be performed. Seven metallographic samples were taken from various regions of the heat pipe. One of them included the failure holes. After polishing, the larger of the two holes was exposed in cross-section. Electron microprobe analysis indicated the presence of nickel in the area adjacent to the hole. Because there is a peritectic in the Mo-Ni binary phase system with a melting temperature of 1635 K, it was concluded that the observed structure was the remnant of a nickel-rich liquid, while areas of lower nickel concentration corresponded to molybdenum-rich Mo-Ni solid solution. Ion microprobe analysis indicated that lithium and oxygen were also present in the region.

Grain boundary penetration was observed in the vicinity of the hole on the inside of the heat pipe along with an occasional string of grain boundary precipitates near the inside surface of the tube. A surface layer found on the lower portion of the evaporator inner surface was analyzed with the electron microprobe, and it was found to be rich in hafnium. Hafnium was also found to have diffused 10 to 20 μm into the heat pipe wall, and in some areas near the inside of the tube wall, hafnium-rich grain boundary precipitates were found at greater depths. No remnant of the hafnium getter foils was found in their original

location after the test; however, single crystals of hafnium carbide were collected off the inside surface of the molybdenum screen.

Chemical analyses of container-tube sections taken from various regions of the heat pipe showed that the carbon and oxygen content near the getter packet were very low, 5 ppm and < 5 ppm, respectively, while in the center of the evaporator carbon was 15 ppm and oxygen 20 ppm. Tube sections from the condenser were found to contain 10 to 20 ppm C and 10 to 45 ppm O. The oxygen concentration in the condenser screen wick near the evaporator end of the condenser was 110 ppm. These analyses indicated a pattern of carbon and oxygen transport to the evaporator and to the extreme end of the condenser.

Interpretation of Analysis

A possible mechanism for the failure of this heat pipe is indicated by the analyses. Stainless steel component impurities, Fe, Ni, and Cr were concentrated in the evaporator of the heat pipe during operation, until the nickel concentration reached a level in excess of 1.8 at.% locally and a liquid phase peritectic formed which eventually penetrated the heat pipe wall. Because other elements, such as Fe, Cr, Si and O, were found in the vicinity of the hole, the actual failure mechanism is probably more complex than this simple model, however as the lithium in this test had been processed and stored in stainless steel containers and not distilled into the heat pipe, it is reasonable to presume it was contaminated with nickel due to the high solubility of nickel in lithium.

The corrosion observed in the Mo/Li heat pipe was related to impurity effects and occurred in the evaporator. The impurities may have been residual in the alkali metal or in the heat-pipe container or wick. The rate of transport of the impurities to the evaporator governed the heat pipe lifetime. Due to the complexity of the corrosion processes in heat pipes much more experimental and theoretical work will be required to completely identify the specific reactions. It is essential that experimental and theoretical work proceed concurrently because neither alone can satisfactorily establish cause and effect relationships in alkali metal heat pipe corrosion.

TRANSIENT HEAT PIPE INVESTIGATION

Test Hardware Description:

A 2.0 m long annular wick heat pipe with a design axial flux of greater than 20 kW/cm^2 was designed and fabricated for use in an extended operation test program at a nominal operating temperature of 1500 K.

The 2 meter long heat pipe employed an annular wick of 400 mesh molybdenum-rhenium screen and a containment tube of 1.59-cm diameter low carbon molybdenum tubing. Wall thickness of the tubing was 0.8 mm with an annulus space of 0.38 mm and a screen wick thickness of 0.30 mm. Interior vapor space area was 1.26 cm^2 . The evaporator

length of the heat pipe was 40 cm with the remainder of the heat pipe serving as condenser. The condenser region of the heat pipe was coated with zirconium diboride by plasma spray to raise the effective emissivity to 0.61 for the radiation loaded test. The resultant axial power level at 1500 K was 14 kW. A cross section of the heat pipe is shown in Figure 2. Power input to the heat pipe was provided by RF induction at approximately 400 khz, and a water cooled coaxial calorimeter served as the heat sink for radiation from the condenser as indicated in Figure 3. Power throughout was determined from water flow and temperature change measurements at the calorimeter. Heat pipe

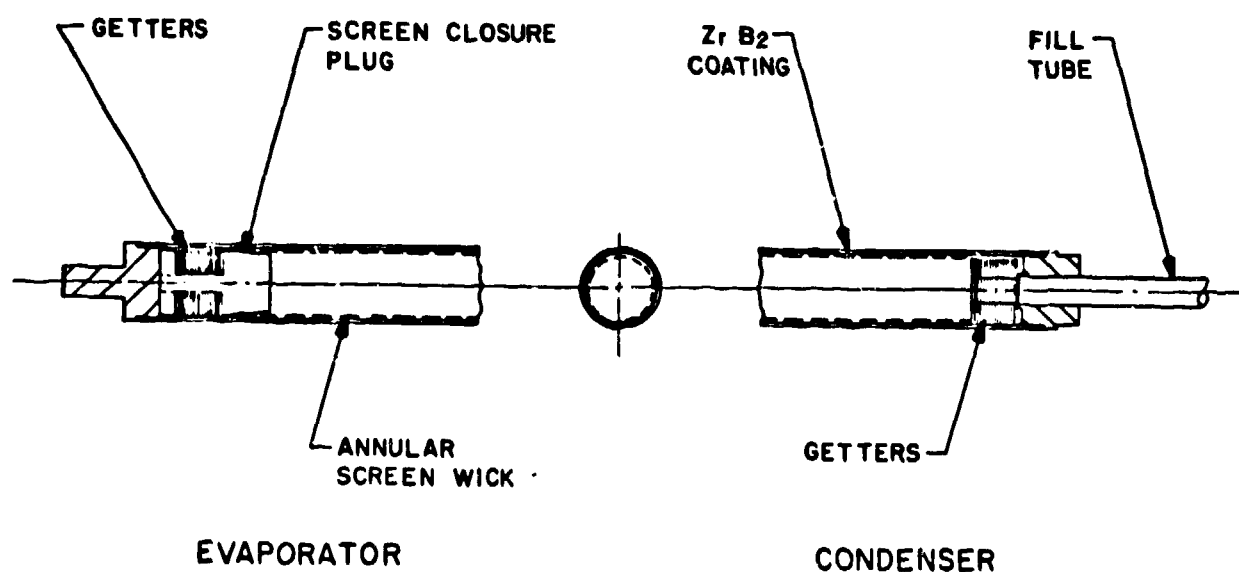


Fig. 2. Cross section of extended operation test heat pipe.

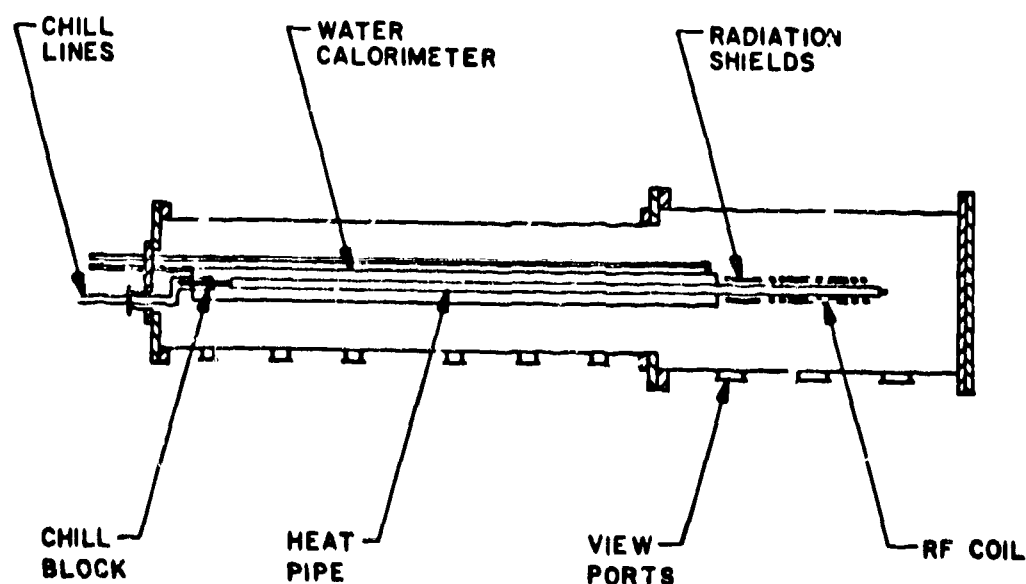


Fig. 3. Heat pipe test set-up.

temperature was measured by tungsten-rhenium thermocouples welded to the heat pipe in the evaporator exit area. The test was conducted in a vacuum chamber maintained at a pressure level of 10^{-6} torr throughout the test.

Test Operation

After filling and wet-in of the heat pipe it was installed in the test chamber and operated at 1400 K for 6 hours for operational verification. The test was then shutdown over a weekend. The following Monday the heat pipe was brought to the extended operation test condition by increasing the RF power to the evaporator over a period of hours. Once the design temperature of 1500 K had been achieved the heat pipe was operated at constant power input. Steady state operation was continued for a period of 514 hours with no apparent change in the heat pipe operating characteristics. At 514 hours the test operation was shut down for planned maintenance of the RF heating system. In the process of reducing the power and temperature of the heat pipe a dry-out of the heat pipe evaporator occurred. Restart attempts led to repeated evaporator dry outs.

The heat pipe was maintained overnight at 1075 K in an attempt to rewet the dried out region of the evaporator without success. The heat pipe was removed from the test chamber for X-ray and neutron radiography to determine the condition of the wick structure and the disposition of the lithium in the annulus. The radiography showed transfer of lithium to the condenser end of the heat pipe leaving the annulus empty in the evaporator region. The X-rays indicated damage to the wick structure in the evaporator. To effect a repair the evaporator end of the heat pipe was opened, the screen tube shortened by 10 cm, and the evaporator end of the screen resealed with a zirconium brazed plug. The annular screen wick was reinserted into the shortened container tube, new getter discs of hafnium and zirconium emplaced outside of the screen closure plug and an end closure electron beam welded in place to reseat the heat pipe. The repaired heat pipe was refilled with lithium, wet-in by operation at low power in multifoil radiation shields and reinstalled in the test chamber.

The test was restarted and the heat pipe operated continuously for an additional 500 hours at 1500 K and 14 kW to complete the originally planned 1000 hour demonstration. At the completion of the test period the heat pipe was removed from the test chamber for a repeat of X-ray and neutron radiography and for general physical inspection. No evidence of further damage to the heat pipe structure was discovered in these examinations. However a void in the lithium fill of the annulus was observed in the evaporator region of the heat pipe.

The heat pipe was then rewet at 675 K for 5 hours in a vacuum furnace, reinstalled in the test chamber, brought to the design operating condition and operated for an additional 744 hours at 1500 K and 14 kW. At this time the test was interrupted by an unplanned transient in the data recording system which triggered an interlock shutdown of the RF

generator. The heat pipe was allowed to cool, removed from the chamber and rewet in a vacuum furnace for 5 hours at 675 K. It was then reinstalled in the test chamber and brought to 1210 K and a power level of about 4.0 kW before the operation was interrupted by a vacuum interlock shutdown. Attempts to restart the heat pipe were unsuccessful. The heat pipe was then removed from the test chamber, and subjected to neutron radiography and X-Ray examination. The radiographs again showed a lithium void in the evaporator annulus, however, the wick did not appear to be damaged. A decision was made at this point to discontinue the extended operation test and investigate the dry out conditions. As a first step operation through shut down and restart at reduced load was investigated. Radiation shields were installed in the calorimeter to reduce the heat pipe load to about 6 kW at 1500 K. The heat pipe was again rewet in the vacuum furnace, reinstalled in the chamber, brought to temperature and operated at 1500 K for 24 hours. At the end of this period the heat pipe power input was shut off, the pipe allowed to cool to about 900 K, input power reapplied, and the temperature brought back to 1500 K over a period of about 2 hours. Next the heat pipe was reduced in power throughput gradually until the temperature was about 1000 K. No operational interruption was seen during either of these transients. The heat pipe was allowed to cool over night and a restart attempted the following day. In start-up the heat pipe again dried out in the evaporator at a temperature of about 1000 K.

Closely fitted radiation shields were next installed in the chamber and the heat pipe successfully brought to 1500 K. Power level under these conditions was about 2 kW. The heat pipe was reduced in temperature under power to about 1000 K and then shutdown. Radiation shields were removed and a restart attempted unsuccessfully. Further furnace wet-in attempts were unsuccessful and radiography showed wick damage similar to that seen at 514 hours.

Discussion of Test Results

Prior to the start of the 1000 hour test the heat pipe was operated at 1400 K with the radiation calorimeter in the test chamber, shutdown, and restarted without incident. The initial dry-out occurred under power and near the sonic limit while reducing the heat pipe temperature after 514 hours of full power operation. This suggests that some deterioration of the wick structure leading to an increased capillary pore radius may have occurred in the first 500 hours of operation. If the wick effective pore size was unchanged at about 50 μm the heat pipe would not have been expected to dry out due to evaporator sub-cooling. Any change in wick characteristics prior to the 500 hour dry-out would have had to be minor, however, as the heat pipe continued to operate at full power and temperature, indicating an effective pore size of ~ 120 to $150\mu\text{m}$. The gross damage observed in disassembly at 514 hours must have occurred after normal operation had been interrupted. The two subsequent dry-outs occurred in restart after a no-load shut down to room temperature. Furnace re-wetting after these dry-outs allowed the heat pipe to be restarted and operated to full

power. Subsequent tests demonstrated that at reduced power levels it was possible to bring the heat pipe from the operating temperature of 1500 K to below sonic limit conditions and back to full power so long as the lithium remained liquid. Finally, the tests with closely spaced radiation shields showed that it was possible to restart the heat pipe from the frozen condition without a furnace re-wet operation if the start-up load on the pipe could be sufficiently reduced. The extremely low vapor pressure (10^{-8} mm) of lithium at the melting point would suggest that if the heat pipe did dry-out in shutdown due to sub-cooling of the condenser it should rewet before solidification of the lithium as the pressure difference within the heat pipe decreased in cool-down. Therefore, the failure of the heat pipe to restart in subsequent tests after the wick structure was repaired was probably due to a different cause than the original dry-out.

Statement of The Problem

Pending further investigation it is assumed that the cause of the restart problem is the formation of voids in the evaporator region of the annulus with local de-wetting of the heat pipe wick during fluid solidification. This is assumed to be caused by directional solidification of the heat pipe working fluid and is perhaps aggravated by an increase in the effective pore size of the heat pipe wick over the operating period. The dry out observed during shutdown under power may have been similarly caused by a change in the wick structure in the prior 514 hours of full power operation. At this point there is no evidence that operational interruption of the heat pipe occurs at the sonic limit point if the shut down is conducted by shutting off the input power.

Problem Resolution

Given the assumptions listed above as to the cause of the restart problem a variety of possible solutions may be considered. Finer pore structure wicks would be more resistant to de-wetting during void formation in the freezing liquid and their use would reduce the magnitude of the problem, however, as the heat pipe was successfully shut down, frozen, and restarted prior to the start of the 1000 hour test it seems that the design value for the wick pore size (50 μ m) will provide sufficient capillary force to prevent the dry out if the wick does not deteriorate during operation. Use of a noncondensable gas has been suggested as a means of reducing start-up and shutdown problems. However, control of noncondensable gas in an artery or annular wick heat pipe in order to ensure that the gas cannot enter the arteries is probably a more difficult problem than alternative methods of liquid inventory control. Artery heat pipes having comparatively thick walled wick structures will provide a larger shrinkage volume of the working fluid prior to de-wetting and will, therefore, be more resistant to dry out than thin annular wicks such as that used in this test heat pipe. Any means of raising the heat pipe temperature uniformly to above the melt temperature of the liquid would eliminate the start-up problem, however, most of the methods suggested for accomplishing this;

such as lower temperature heat pipes, electrical heaters, or variable radiation shields would greatly complicate a heat pipe heat transport system. The simplest potential solution to the problem is to include a liquid reservoir in the evaporator end of the heat pipe with sufficient surplus liquid provided to accomodate shrinkage during solidification. As a test of this potential solution the present test heat pipe has been modified to incorporate such a reservoir wick structure and is being prepared for re-test. The results of these further tests will serve to much more closely define the problem and the method of resolution.